Semisolid Magnesium Feedstock Produced by Controlled Nucleation Method

H. Wang[1] and Z. Ning[1]

[1] School of Engineering, The University of Queensland, St Lucia, QLD 4072, Australia
[2]CRC for Cast Metals Manufacturing (CAST), UDP No.055, St Lucia, QLD 4072, Australia
[3]CSIRO Manufacturing Science and Technology, PO Box 883, Kenmore, QLD 4069, Australia

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Abstract

A Controlled Nucleation method has been developed to produce feedstock for semisolid forming, without forced convection. By controlling grain nucleation and growth, fine-grained and non-dendritic microstructures that are suitable for semisolid casting can be generated. The method has been applied to hypoeutectic and hypereutectic aluminium-silicon casting alloys, aluminium wrought alloys and a magnesium alloy. Parameters such as pouring temperature, cooling rate and grain refiner addition have been controlled to achieve copious nucleation, nuclei survival and dendritic growth suppression during solidification. The influences of the controlling parameters on the formation of thixotropic structure are different for each of these alloy groups. The as-cast structures were then partially remelted and isothermally held. Semisolid thixotropic structures were developed and ready for semisolid casting.

Introduction

Semisolid metal forming (SSMF) is an emerging technology for near net-shape production of engineering components, in which metal alloy is processed at a temperature between its solidus and liquidus temperatures [1,2]. The microstructure of material suitable for SSMF typically has globular solid particles uniformly dispersed in a liquid matrix [3]. Such structures provide unique flow behaviour when sheared during the forming process [4], with minimal surface turbulence and splashing, and this smooth die filling assists in producing good microstructural integrity for the final products.

To develop this structure, loosely termed a thixotropic structure, external convection or shear is often applied during solidification of the melt, preventing growth with a dendritic morphology. Several methods have been reported to successfully produce the thixotropic structure in aluminium and magnesium alloys using forced convection. These include mechanical stirring [5], electromagnetic stirring [6], ultrasonic stirring [7] and, most recently, melt mixing [8]. The external convection methods usually involve extra equipment and complicated operation. This results in a significant cost increase for the feedstock, which is one of the main reasons that temper a wider application of SSFM. It has become more and more important to develop a simple, practical and less expensive method to produce the semisolid thixotropic structure.

An alternative to the external forced convection is solidification control. During solidification, dendritic growth is affected by parameters such as nucleation density, grain growth, solute redistribution, ripening and interdendritic fluid flow. The Controlled Nucleation method uses the control of certain solidification conditions, such as pouring temperature, cooling rate and grain refinement, to maximise grain nucleation and suppress dendritic growth, thereby producing a starting microstructure that is fine-grained and less dendritic. Such microstructures can evolve to a globular structure after partial remelting and isothermal holding, which is usually employed prior to the semisolid casting. A number of methods have been reported that rely on a Controlled Nucleation method; such as liquidus casting [9], grain refinement [10], New Rheocasting [11] and SSR process [12]. In this study, the Controlled Nucleation method is applied to aluminium foundry alloys (hypoeutectic and hypereutectic aluminium-silicon alloys), aluminium wrought alloys and a magnesium alloy. This study examines the relative effectiveness of each nucleation control mechanism in the different alloy systems, on the formation of thixotropic structure.

Experimental Procedures

The alloys used in this study were hypoeutectic aluminium-silicon alloy A356, hypereutectic aluminium-silicon alloy A390, aluminium wrought alloy 6063 and magnesium alloy AZ91. Their chemical compositions are given in Table 1. The A356, 6063 and AZ91 were commercial ingots. Alloy A390 was produced in-house using 99.7% Al. The Al alloys were melted in an induction furnace and degassed. Al-5Ti-B master alloy was used as a grain refiner for alloy A356 and alloy 6063. AlP-ready master alloy ALCUP was used in A390 alloy for primary silicon phase refinement. AZ91 was charged in a dedicated electric resistance furnace under the protection of SF_6 cover gas. The melts were cast into cylindrical steel moulds, a cylindrical cavity of 50 mm in diameter and 70mm deep for a lower cooling rate and a cylindrical cavity of 20 mm in diameter and 200 mm deep for a higher cooling rate. Various pouring temperatures were used to give a range of superheats. The mould preheat temperature was 200°C.

Table I. Chemical compositions of alloys used (in wt %).

Alloy	Si	Mg	Cu	Mn	Ti	Fe	Zn	Al
A356	6.8	0.36	<0.01	<0.01	0.015	0.08		Balance
A390	16.8	0.7	4.6	<0.01	0.01	0.09		Balance
6063	0.40	0.61	0.02	<0.01	<0.005	0.13		Balance
AZ91	0.03	Balance	0.005	0.33		0.001	0.65	9.03

In order to test the contribution made to nucleation by wall crystals, some gauze experiments were carried out for the Al-based alloys. A stainless steel gauze was placed in the centre of the mould and melt was poured inside the gauze. The gauze served as a mechanical barrier for the melt movement from the mould wall to the centre, so any wall crystals formed on or near the wall would be isolated between the wall and the gauze.

Small cylindrical samples ($\Phi 20 \times 10$ mm) were cut from the castings. For aluminium alloys, the samples, after preheating, were placed in a molten salt-bath for partial remelting and isothermal holding. The heating rate was about 15°C/s. For magnesium alloys, the samples were placed in an electric resistance furnace with the protection of SF_6. A large steel block was used as a heat reservoir. The isothermal holding was carried out at a temperature in the semisolid region determined from the phase diagram of each alloy. The samples were then quenched in iced water for microstructural examination. As-cast samples and partially remelted samples were sectioned and polished. The polished samples were anodised or etched. The microstructures of the samples were examined using optical microscopy.

Results

Magnesium alloy AZ91

Figure 4 shows the as-cast microstructures and semisolid evolved microstructures of magnesium alloy AZ91. Semisolid isothermal holding was carried out at 575°C for 10 minutes.

Figure 4a shows the microstructure of the sample cast at higher superheat temperature (125°C). It consisted of primary α-Mg phase and divorced eutectic β-phase ($Mg_{17}Al_{12}$). The primary Mg phase was of dendritic morphology with a grain size of 360 μm. After partial remelting the primary phase became very irregular, and grains appeared separate from each other. The structure was not suitable for semisolid processing. Figure 4c shows the microstructure poured at a lower superheat (10°C). The primary Mg phase was still of a dendritic morphology but the dendrites became much less distinct and the grain size was reduced to about 70 μm. After the semisolid remelting, the microstructure became very globular with a particle size of about 100 μm (Figure 4d). It seemed to be an ideal structure for semisolid processing. Figure 4e show the microstructure from the same superheat but at a higher cooling rate. The higher cooling rate resulted in a more rosette-like structure but the grain size remained similar. The semisolid structure was also similar to the low cooling rate sample, as shown in Figure 4f.

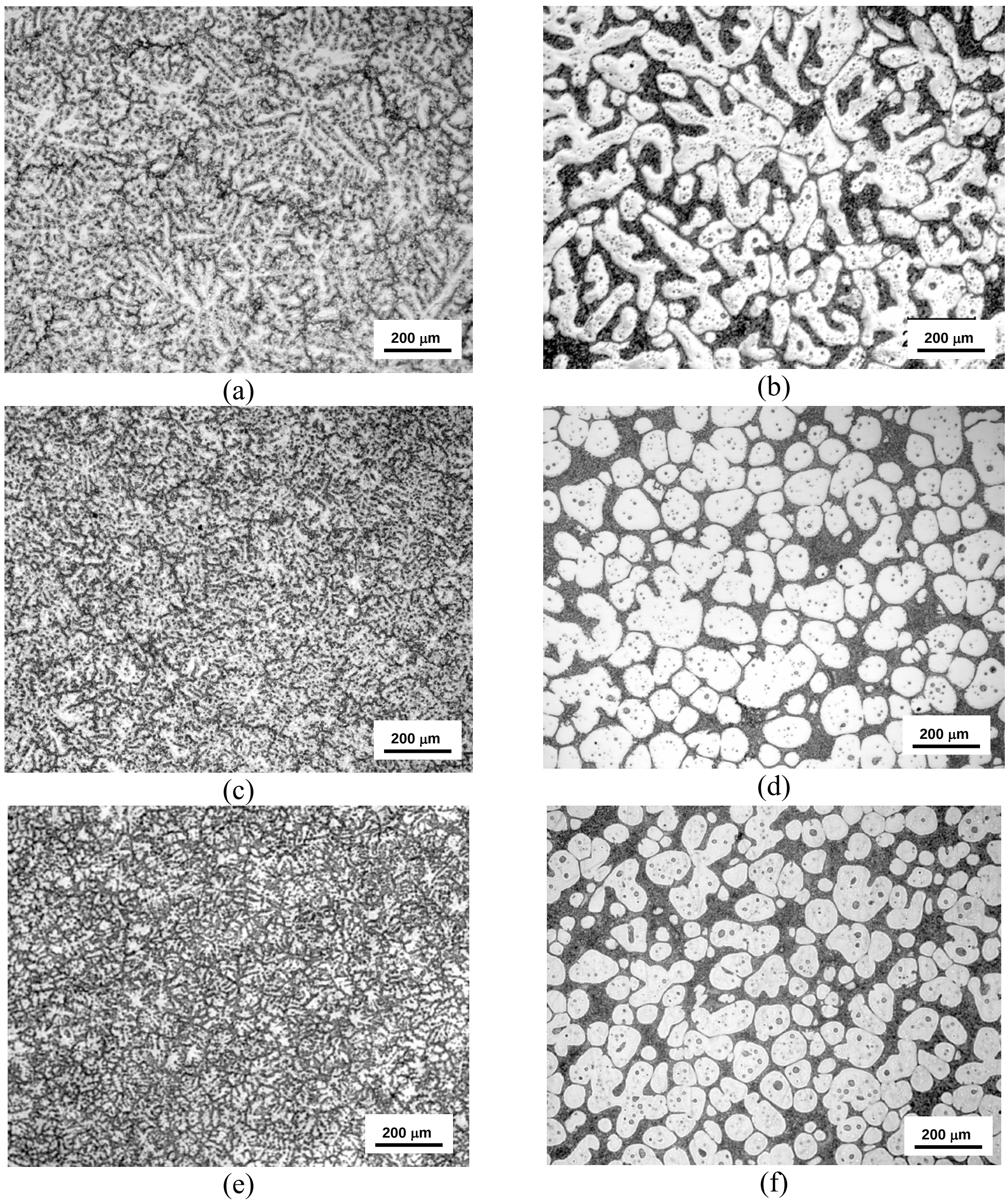

Fig.4. Microstructures of alloy AZ91. (a), (c) and (e) are as-cast microstructures; (b), (d) and (f) are semisolid microstructures after isothermal holding at 575°C for 10

mins. The casting conditions are: (a) and (b) high superheat, low cooling rate; (c) and (d) low superheat, low cooling rate; and (e) and (f) low superheat, high cooling rate.

Discussion

In the Controlled Nucleation method, the key points are copious nucleation, nuclei survival and suppression of dendritic growth. The main sources of grain nuclei during solidification are the grain refiner, which supplies heterogenous particles, and detached wall crystals. Constitutional undercooling controls the activation of nuclei and is strongly affected by the solute content in the alloy, which will also influence the consequent grain growth mode. However an increased alloy content, which promotes constitutional undercooling, also promotes a more dendritic morphology. Grain refiner addition can be very effective for some alloys, but less successful in others, such as Mg(Al) alloys. Wall crystals provide another nucleation source that can be exploited. Wall crystals are the crystals that are nucleated during pouring, at or near the relatively cold mould wall. They are then carried to the bulk of the melt by fluid flows within the melt and serve as very effective nuclei. Furthermore, dendrite arm fragments may melt off, providing multiple nuclei from a single original source. The larger the number of wall crystals generated, transported and that survive in the melt, then a finer grain structure with less dendritic morphology will be produced. Parameters such as pouring temperature, cooling rate and other mould thermal conditions can be controlled to promote these nucleation mechanisms and achieve the maximum nucleation density. It is possible to adjust grain nucleation and growth to produce a starting microstructure that is fine-grained and less dendritic. Followed by a partial remelting and isothermal holding, which is usually employed prior to the semisolid casting, such microstructures can evolve to a globular structure. The effects of these controlling parameters on the formation of thixotropic structure and the contribution of each nucleation mechanism are different for the different alloy systems. These experiments have shown that there are substantial differences between alloy systems in the relative contributions from the different mechanisms.

In the hypoeutectic alloy A356, wall crystals made a significant contribution to the grain structure formation. As shown in Figure 1, the grains inside the gauze exhibited very coarse grains, on the other hand, samples without gauze had a very fine microstructure. The low pouring temperature was further beneficial for the wall crystal mechanism as the survival rate of the wall crystals would be much higher at a low melt temperature. Grain refinement was very effective in high temperature pouring, however without the wall crystal mechanism, it was still not sufficient to generate a thixotropic structure (Figures 1d). The combination of wall crystals and grain refinement resulted in a fine-grained rosette-like structure, which evolved to a globular thixotropic structure with a particle size of 100 μm after remelting (Figure 1h).

In the hypereutectic alloy A390, the coexistence of primary silicon and primary aluminium was important for the formation of thixotropic structure because the amount of primary silicon by itself was insufficient for semisolid forming. The microstructure of the primary silicon structure was effectively controlled by AlP refinement and refinement due to the solidification rate. Wall crystals did not assist in improving the microstructure of A390. A uniform thixotropic semisolid structure was produced with Si particles at a size of 20 μm and Al particles at a size of 60 μm.

In the aluminium alloy 6063, grain refinement was very effective in generating the thixotropic structure. It changed a very coarse columnar structure to extremely fine-grained structure with a grain size of only 50 μm. The wall crystal mechanism was also effective, producing, in the absence of grain-refiner, a globular structure with a particle size of 300 μm.

In magnesium alloy AZ91, the wall crystal mechanism promoted by low temperature pouring was effective. Cooling rate was less effective, at least in the low superheat material, at producing a finer structure, however it led to a more globular grain morphology. A very globular structure with a particle size of about 100 μm was produced (Figures 4d and f).

Conclusions

1. The Controlled Nucleation method uses the control of nucleation and grain growth to produce fine-grained and less dendritic structures. Such structures can evolve to a thixotropic structure after semisolid remelting, ready for semisolid forming.
2. Microstructures suitable for semisolid processing can be produced for each of the alloys studied, but the dominant nucleation mechanism varies between the alloys.
3. The main mechanisms responsible for formation and activation of grain nuclei are wall crystal formation, grain refinement and constitutional undercooling. Solidification parameters such as pouring temperature, cooling rate and grain refiner addition can be controlled to maximise the nucleation density.
4. In the hypoeutectic alloy A356, wall crystals and grain refiner have similar influences on the grain structure. In hypereutectic A390, AlP refinement and thermal refinement are effective, while wall crystals had negligible influence. In aluminium alloy 6063, Ti-B grain refinement is extremely effective, rather more so than the contribution from wall crystals. In magnesium AZ91, the wall crystal formation mechanism is the most effective mechanism.

References

1. M.C. Flemings, "Behaviour of Metal Alloys in the Semisolid State", *Metall. Trans.*, 22A (1991), 957-981.
2. D.H. Kirkwood, "Semisolid Metal Processing", *Inter. Mater. Rev.*, 39 (1994), 173-189.
3. H. Wang, D.H. StJohn, C.J. Davidson, and M.J. Couper, "Characterisation and Shear Behaviour of Semisolid Al-7Si-0.35Mg Alloy Microstructures", *Aluminium Transactions*, 2 (2000), 57-66.
4. H. Wang, C.J. Davidson, and D.H. StJohn, "Semisolid Casting of A356 Alloy Produced by Controlled Pouring", *1st Inter. Conf. on Light Metals Tech.*, ed. A. Dahle, Australia, 2003, 219-222.
5. Z. Fan, "Semisolid Metal Processing", *Inter. Mater. Rev.*, 47 (2002), 49-85.
6. C.Pluchon, W.R. Loue, P.Y. Menet, and M. Garat, "Development of Semisolid Metal Forming Feedstock and Finished Parts", *Light Metals*, (1995), 1233-1242.
7. V.O. Abramov, O.V. Abramov, B.B. Straumal, and W. Gust, "Hypereutectic Al-Si Alloys with a Thixotropic Structure Produced by Ultrasonic Treatment," *Materials & Design*, 18(1997), 323-326.
8. J.L. Jorstad, D. Apelian, and M.M. Makhloul, "Novel, Slurry-based, Semisolid Processing Routes", *Proceedings of 1st Inter. Conf. on Light Metals Tech.*, ed. by A. Dahle, Australia, (2003), 109-114.
9. K. Xia, and G. Tausig, "Liquidus Casting of a Wrought Aluminium Alloy 2618 for Thixoforming", Mater. Sci. Eng., 246A (1998), 1-10.
10. G. Wan, T. Witulski, and G. Hirt, "Thixoforming of Aluminium Alloys Using Modified Chemical Grain Refinement for Billet Production", *La Metallurgia Italiana*, 86 (1994), 29-36.
11. H. Kaufmann, and P.J. Uggowitzer, "The Latest Development of New Rheocasting for Al- and Mg-Alloys", *1st Inter. Conf. on Light Metals Tech.*, ed. by A. Dahle, Australia, (2003), 115-119.
12. J. Yurko, R.A. Martines, and M.C. Flemings, "Development of the Semi-solid Rheocasting (SSR) Process", *the 7th Inter. Conf. On the Processing of Semi-Solid Alloys and Composites*, Tsukuba, Japan (2002), 659-664.
13. H. Wang, Z.L. Ning, C.J. Davidson, and D.H. StJohn, "Thixotropic Structure Formation in A390 Hypereutectic Al-Si Alloy", This Conference.